

The Effect of Nitrogen on the Tensile and Creep-Rupture Properties of Niobium

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ABSTRACT

Variations in the mechanical properties of niobium caused by nitrogen additions in amounts to give single-phase and two-phase structures were investigated at both room and high temperatures. Nitrogen in solid solution raised room-temperature hardness and tensile strength. Increasing the nitrogen content to give two-phase structures however produced no additional strengthening. At elevated temperatures, the effect was different in that, with increase in nitrogen content, there was a continued rise in tensile strength through the two-phase region.

Creep-rupture strength of niobium was improved by nitrogen in solution at temperatures slightly below 0.5 times the melting temperature, whereas above this temperature the strength was unaffected. This change in the nitrogen effect with temperature appears to be related to a concurrent change in the mechanism controlling creep. An increase in the nitrogen content of the two-phase alloys produced strengthening throughout the 1700° to 2600°F test range. Ductility was high for all alloys at elevated temperatures and, except for a high-nitrogen alloy, also at room temperature.

PROBLEM STATUS

This is an interim report on one phase of a continuing problem.

AUTHORIZATION

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THE EFFECT OF NITROGEN ON THE TENSILE AND CREEP-RUPTURE PROPERTIES OF NIOBIUM

INTRODUCTION

In studies of the properties of niobium, it has been observed that small amounts of oxygen and nitrogen in solid solution strengthen the metal in the vicinity of room temperatures. The effects have been noted to extend to elevated temperatures, in the strain-aging region, but are presumed, on the basis of dislocation-locking theory, to essentially vanish at higher temperatures. Contrary to expectations, a previous study (1) showed that oxygen improves creep-rupture strength even in the range 1900° to 2600° F. Available data on the influence of nitrogen are limited however to temperatures below this range. It has been reported that tensile strength is raised in the 1100° to 1550° F range by nitrogen in solid solution (2) and at 1800° F by 0.29 percent nitrogen, an amount which should yield a two-phase structure (3).

These data on the influence of nitrogen were from exploratory studies; consequently, a complete knowledge of the effects over a wide range of conditions including creep-rupture is lacking. Inasmuch as nitrogen has been reported (4,5) to be a more effective strengthener of niobium at room temperature than oxygen, it would be of interest to ascertain whether nitrogen also strengthens at high temperatures and to examine the magnitude and extent of its effect on tensile and creep-rupture properties.

This investigation was undertaken to determine the influence of nitrogen on mechanical properties of niobium, primarily in the range 1700° to 2600° F. Nitrogen additions up to 0.46 weight percent were made which encompassed both single-phase and two-phase structures. Room-temperature tensile properties also were examined for possible correlation with an observed anomalous decrease in hardness as a function of nitrogen content and for brittleness that might have been produced by the nitrogen.

EXPERIMENTAL METHODS

Specimen Preparation

Electron-beam melted niobium containing less than 90 ppm total of interstitial elements was used in this study; the chemical composition is given in Table 1. Nitrogen and oxygen were analyzed by the Kjeldahl and vacuum-fusion methods, respectively. An ingot of 1-1/2-in. diameter was cold swaged to 3/8-in. diameter before being machined into tensile and creep-rupture specimens of 0.25-in. diameter and 1.0-in. gage length. A subsequent anneal in a vacuum of 10^{-6} torr at 2800° F for 29 hours produced an average hardness of 59 Vickers Hardness No. (VHN) (10-Kg load) and a grain diameter of 0.60 mm. Control rods of 1/4-in. diameter were processed along with the test specimens for checking the contaminant pickup, hardness, and structure.

Nitrogen was added to the niobium in a recrystallized alumina tube furnace at 2300° F for various lengths of time up to a maximum of 112 hours, using a prepurified grade nitrogen gas which was passed through a purification train. Test specimens were prepared containing nitrogen at approximate levels of 0.05, 0.12, 0.24, 0.37, and 0.46

Table 1
Impurity Contents of Niobium

Element	Content (wt.-%)
N	0.0045
O	0.0026
H	<0.0005
C	<0.0010
Ta	<0.05
Zr, W, Hf each	<0.01
Fe, Si, Ti each	<0.005
Cu, Cr each	<0.004
V, Mn, Sn, Mo, Pb, Ni, Mg, Co, Al each	<0.002
B, Cd each	<0.0001

weight percent. The nitrided material was then homogenized by heating in vacuum at 2700°F for 24 hours. Shielding the specimens with niobium foil during these treatments aided in keeping the contaminant pickup to a very low level. Oxygen pickup during the nitridation and homogenization treatments usually totaled less than 30 ppm, but in several instances was almost 50 ppm. Cross sections of the control rods were uniform in hardness, indicating that the nitrogen was equally distributed. Prior to testing, the nitrided specimens were chemically etched to remove remnants of nitride surface layers that might have been remained after the homogenization treatment.

The presence of a single-phase or two-phase structure in the niobium-nitrogen alloys at the test temperatures was established by metallographic examination of specimens which had been encapsulated and then quenched from these temperatures. The structural data agree with the information in the literature on the terminal solubility of nitrogen in niobium (6,7).

Test Procedures

Tension tests were conducted in an Instron Tester at room temperature, as well as at 1900° and 2600°F in a vacuum of 10^{-5} torr. For the tension tests, heating was done by electron bombardment of the specimen. Tests were run at a strain rate of 0.05 in./min, except for the region up to 0.6% strain at room temperature, where the rate was 0.005 in./min. Nitrided specimens earmarked for room-temperature testing were slow cooled from 2700°F to about 1100°F at approximately 275°F per hour.

Creep-rupture tests were performed at 1700° to 2600°F in constant-load machines at vacuums of 10^{-6} torr or better for durations up to 2100 hours. In tests at 1700°F, and in most tests at 1900°F, the specimens were enclosed in an Inconel tube which was heated externally by a resistance furnace. Specimens were protected from desorbed gases by enclosing them in a sheath of niobium foil. No significant pickup of contaminants occurred as indicated by weight-gain measurements. Strain measurements were made by means of a dial gage attached to the pull rod and also an autographic recorder connected to the lever arm. Some tests at 1900°F and all tests at 2300° and 2600°F were conducted

in a cold-wall vacuum furnace equipped with tantalum heating elements. Strains were measured by means of an extensometer attached to the specimen.

The usual practice in loading the creep machines was to apply the full load rapidly at the start of a test. Since unusual scatter in rupture life of unalloyed niobium occurred at 1700° and 1900°F, loading at these temperatures was done slowly in small increments in an attempt to get better reproducibility.

RESULTS

The properties examined in determining the effects of nitrogen at elevated temperatures were tensile strength, rupture life, minimum creep rate, and elongation at fracture. Although behavior at high temperature was the primary interest of the investigation, mechanical property measurements were made also at room temperature to reveal associated structural changes in the material and to examine for possible low-temperature brittleness.

Room-Temperature Properties

Hardness — Nitrogen in solid solution substantially increases the room-temperature hardness of niobium, while nitrogen in larger amounts which yield two-phase structures gives no additional increase. In fact, two-phase structures may be softer than single-phase structures, as illustrated by the hardness impressions on the incompletely homogenized specimen in Fig. 1. The hardness of the single-phase core (210 VHN) is noticeably greater than that of the two-phase surface layer (108 VHN). In contrast, the hardness of unalloyed niobium is approximately 60 VHN.



Fig. 1 - Indentations showing lower hardness of the two-phase region as compared with the single-phase region of nitrified niobium. Etched in HF, HNO₃, and H₂O (1:8:2). 100X.

Similar hardness correlations have been observed between the single-phase and two-phase structures of the homogenized niobium alloys. Specimens quenched from the test temperatures revealed, as shown in Fig. 2, a pronounced increase in hardness with rise in nitrogen content in the single-phase region but no appreciable hardness change in the two-phase region. The observed structures conform to the phase diagram drawn on the basis of data in the literature (6,7). In other specimens that were slow cooled from the homogenization temperature, a peak in hardness was attained in the single-phase material of 0.05 percent nitrogen, while in the two-phase materials the hardnesses were less than the peak value (Fig. 3). Hardness measurements (using a 25-gram load) were made separately on the nitride particles and the matrix of the Nb-0.37% N material slow cooled from 2700°F, the structure of which is shown in Fig. 4. The nitride has a hardness of approximately 1000 VHN, and the matrix hardness, though the values were somewhat inconsistent, averaged 130 VHN, which was lower than the overall hardness of the material (144 VHN).

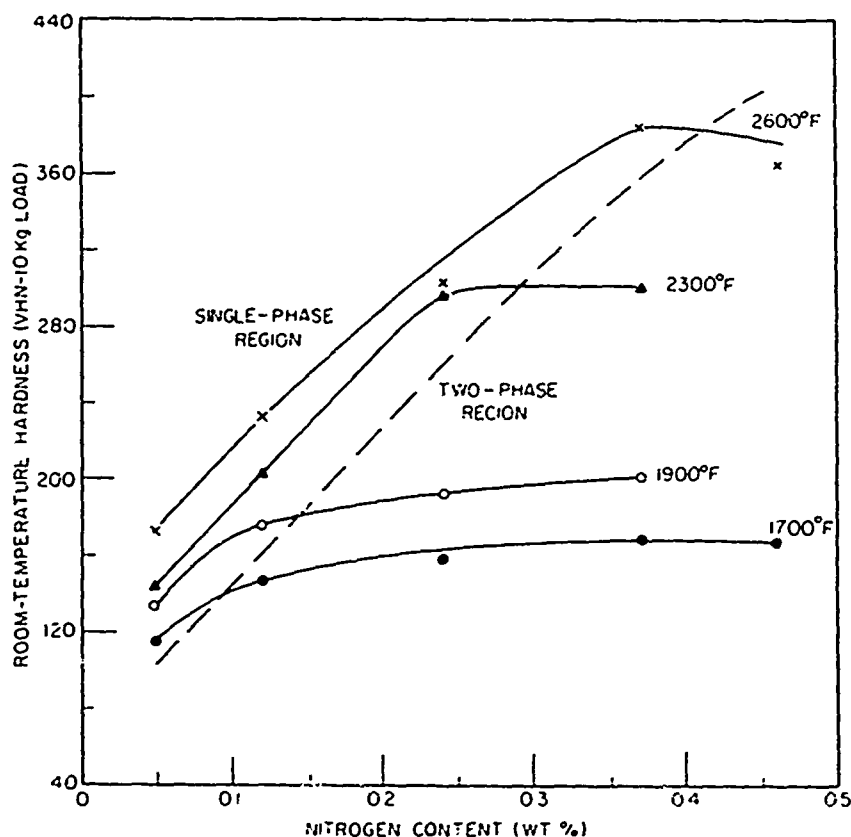


Fig. 2 - Influence of nitrogen on the hardness of niobium-nitrogen alloys quenched from various temperatures in the range of 1700 to 2600 F

Tensile Strength — In conformity with the hardness results, the tensile data also show that nitrogen in solid solution is a very effective strengthener of niobium (Fig. 3). No additional improvement in tensile strength is obtained by the introduction of a second phase in the structure. Actually at the higher nitrogen levels, the strength is lower than the peak strength, though it still remains higher than that of unalloyed niobium. The single-phase and two-phase regions in Fig. 3 were established on the basis of metallographic examination. Changes in yield strength generally corresponded with those in tensile strength except for the 0.37 percent nitrogen material, where the yield and tensile strengths tended to converge.



Fig. 4 - Microstructure of niobium-nitrogen alloys slow cooled from 2700° F: (top) Nb-0.24% N; (bottom) Nb-0.37% N. Etched in HF, HNO₃, and H₂O (1:4:4). 100X.

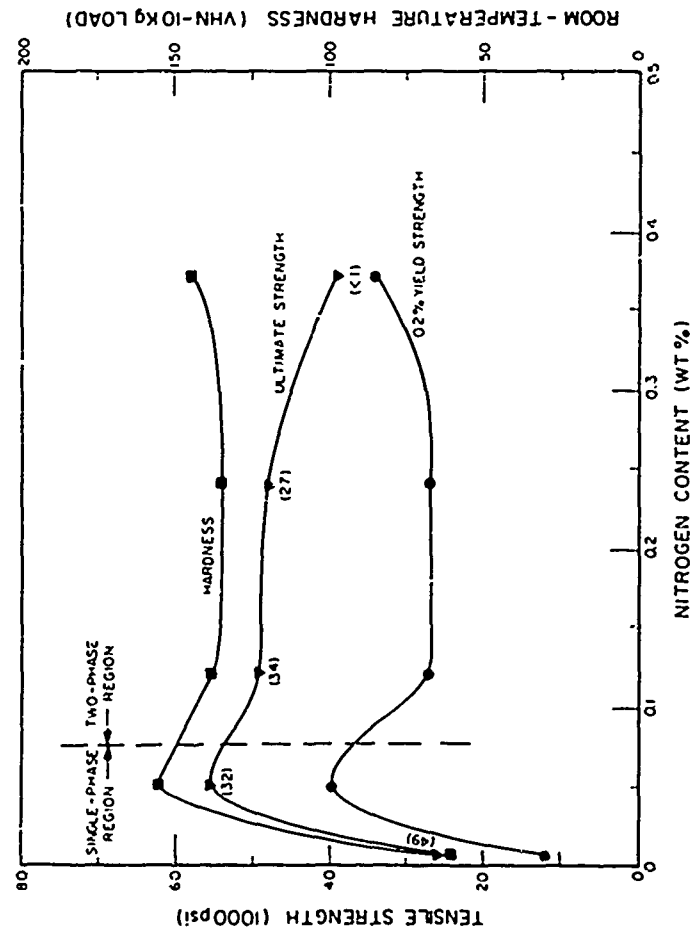


Fig. 3 - Influence of nitrogen on the room-temperature hardness and tensile strength of niobium slow cooled from 2700° F. (Numbers in parenthesis are elongation-at-fracture values.)

Nitrogen, up to 0.24 percent, whether in single-phase or two-phase structures, caused no appreciable loss of ductility; the minimum elongation at fracture was 27 percent. However, the material containing 0.37 percent nitrogen was brittle with an elongation of approximately 1 percent. The pronounced brittleness in the high-nitrogen alloy may be attributed in part to the massive nitrides at the grain boundaries which formed upon slow cooling (Fig. 4). By using a faster rate of cooling, the massive nitrides were avoided, to a large degree, and the elongation rose to 7 percent.

Modulus of elasticity measurements ranged from 15.5 to 18.5 million psi. No consistent trend according to composition of the materials was evident that could account for the variations in these values.

Elevated Temperature Properties

The influence of nitrogen on mechanical properties at elevated temperatures was found to depend principally on phase structure and test temperature, i.e., whether the latter was below or above one-half the absolute melting temperature ($0.5 T_m$). Accordingly, the following generalizations may be made of the results. Nitrogen in solid solution raises the strength of niobium at temperatures below approximately one-half the melting temperature, but at higher temperatures it generally has no significant effect. When the nitrogen concentration is such that the structure is two-phase, then strength at all test temperatures increases with increasing nitrogen content. In the area near the boundary between the single-phase and two-phase regions, a sharp transition in the strength of the material generally occurs which may be either an increase or a decrease in strength.

Tensile Strength - Tensile strength at 1900°F, which is approximately one-half the melting temperature of niobium, was improved with increasing nitrogen content in both the single-phase and two-phase regions (Fig. 5). At 2600°F, solid-solution strengthening occurred only for amounts above 0.24 percent nitrogen. At this temperature, the two-phase alloys, similar to their behavior at 1900°F, were somewhat stronger than the single-phase alloys.

Creep-Rupture Properties - Creep-rupture properties were affected by nitrogen in a consistent way, the magnitude of the effect being dependent upon the test conditions. Nitrogen in solid solution increased rupture life at 1700°F, below $0.5 T_m$, as shown in Fig. 6, but above the $0.5 T_m$ level, at 2300° and 2600°F, it had no distinct effect, Figs. 7 and 8. At 1900°F, which is approximately at $0.5 T_m$ of niobium, both effects were observed (Fig. 9). Nitrogen in solution had no significant influence on rupture life at 6300 psi, but at 6600 psi it markedly increased strength. The effect of nitrogen at the higher stress was the same as that below $0.5 T_m$, while the effect at the lower stress corresponded to that above $0.5 T_m$. When present in a two-phase structure, nitrogen in increasing amounts substantially improved rupture strength at all test temperatures (Table 2).

When solid solution strengthening occurred, as at 1700° and 1900°F, an abrupt reduction in rupture life took place in the region of the boundary between the single-phase and two-phase structures, as shown in Figs. 6 and 9. Although the two-phase alloy in the region of the boundary showed a lower rupture life than the single-phase material, its strength nevertheless was higher than that of pure niobium.

Although there was no strongly discernible effect of nitrogen in solid solution at 2300° and 2600°F, the rupture life of the alloy nearest the phase boundary tended to be the lowest. However, in most cases the apparent decrease in life did not appear to be outside the scatter range of the data, so that this is not considered to be a definite effect. The transition from a single-phase to a two-phase structure at these temperatures was marked by a substantial increase in rupture life, contrary to the accompanying decrease in rupture life at the phase boundary at the lower temperatures.

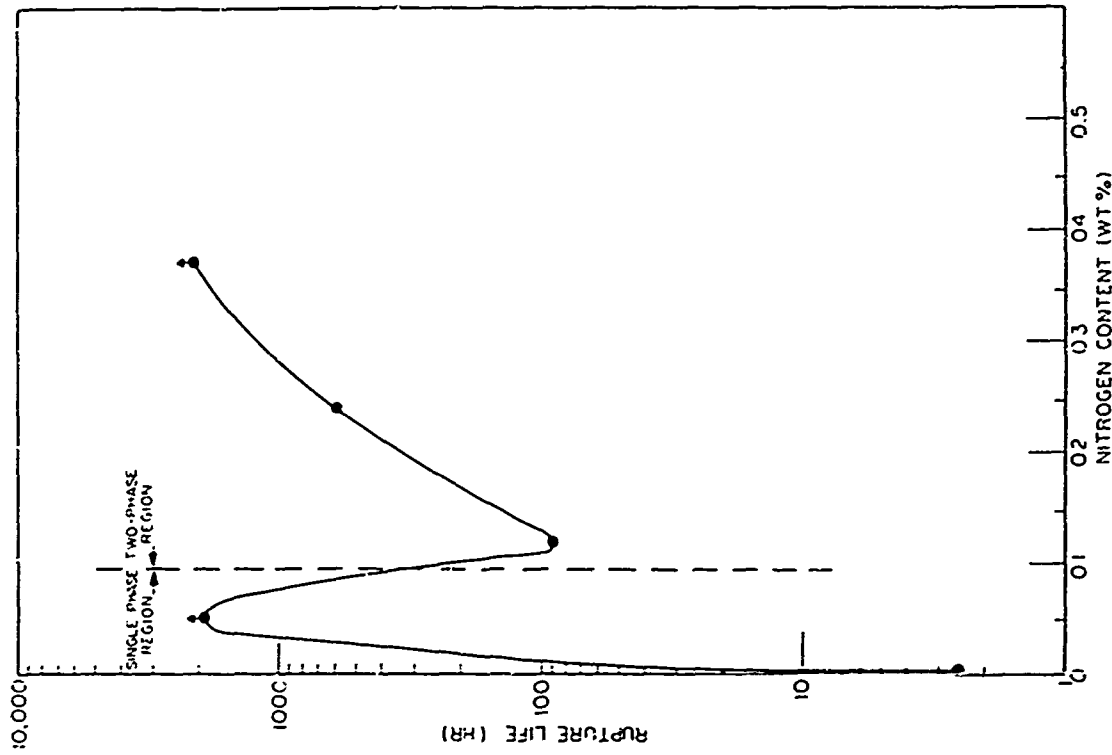


Fig. 6 - Influence of nitrogen on the rupture life of niobium at 1700°F and 6500 psi. (♦ denoted interrupted test.)

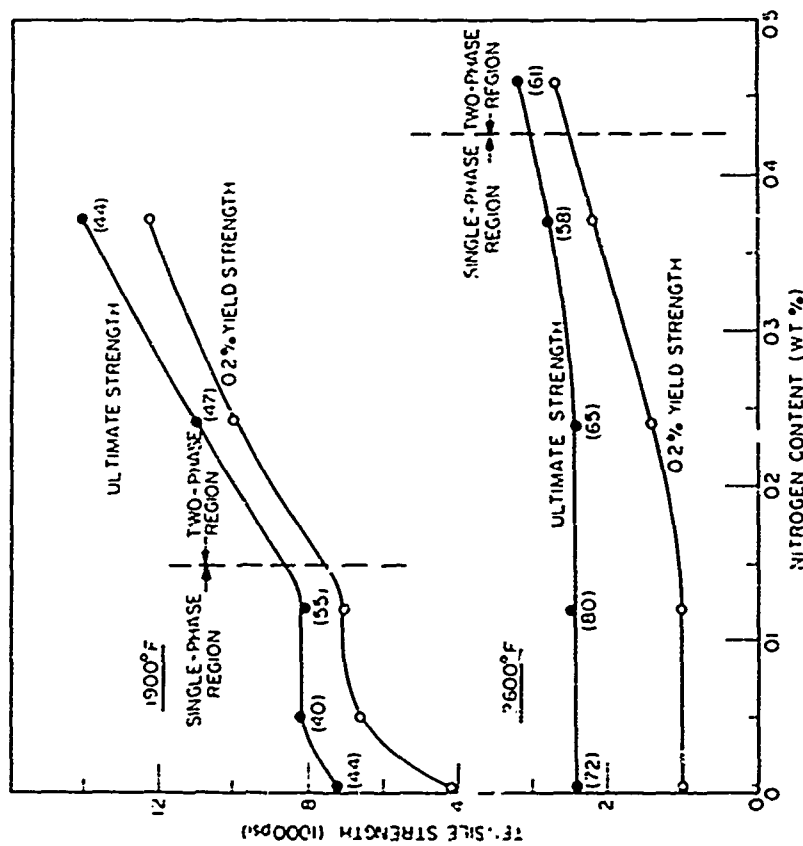


Fig. 5 - Influence of nitrogen on the tensile strength of niobium at 1900°F and 2600°F. (Numbers in parentheses are elongation-at-fracture values.)

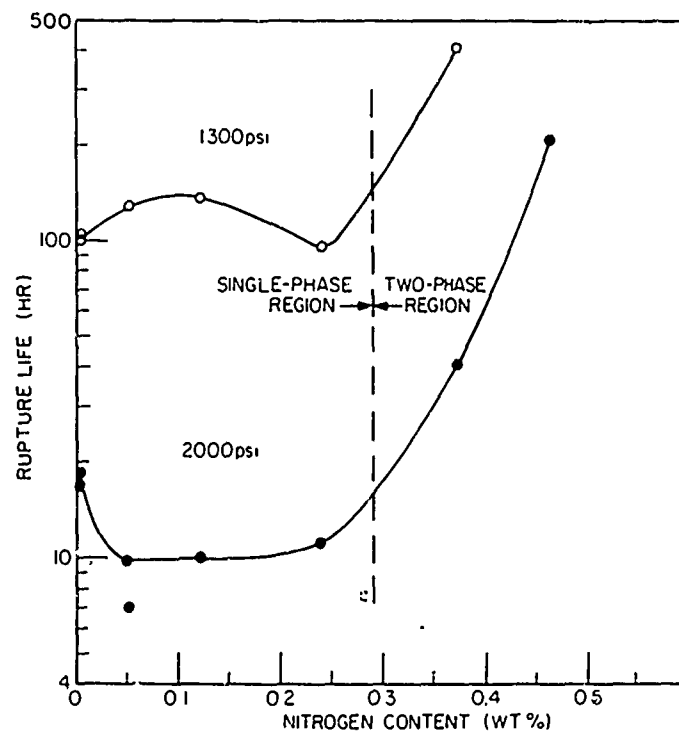


Fig. 7 - Influence of nitrogen on the rupture life of niobium at 2300° F

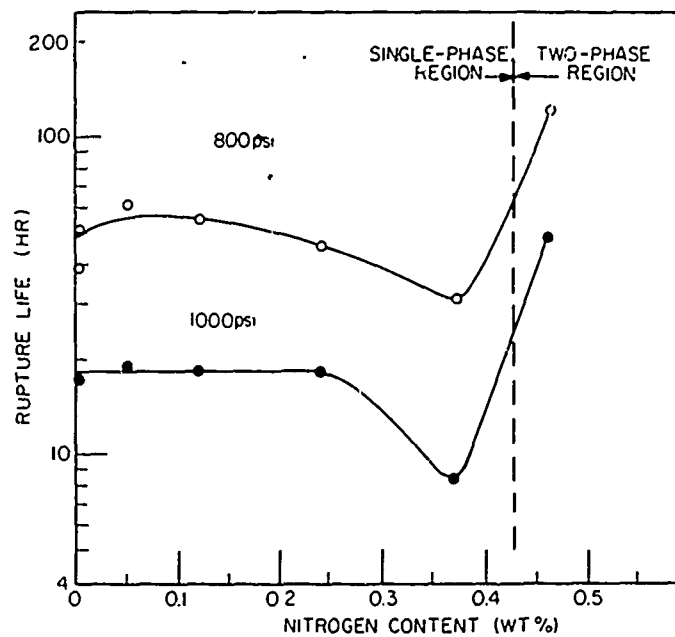


Fig. 8 - Influence of nitrogen on the rupture life of niobium at 2600° F

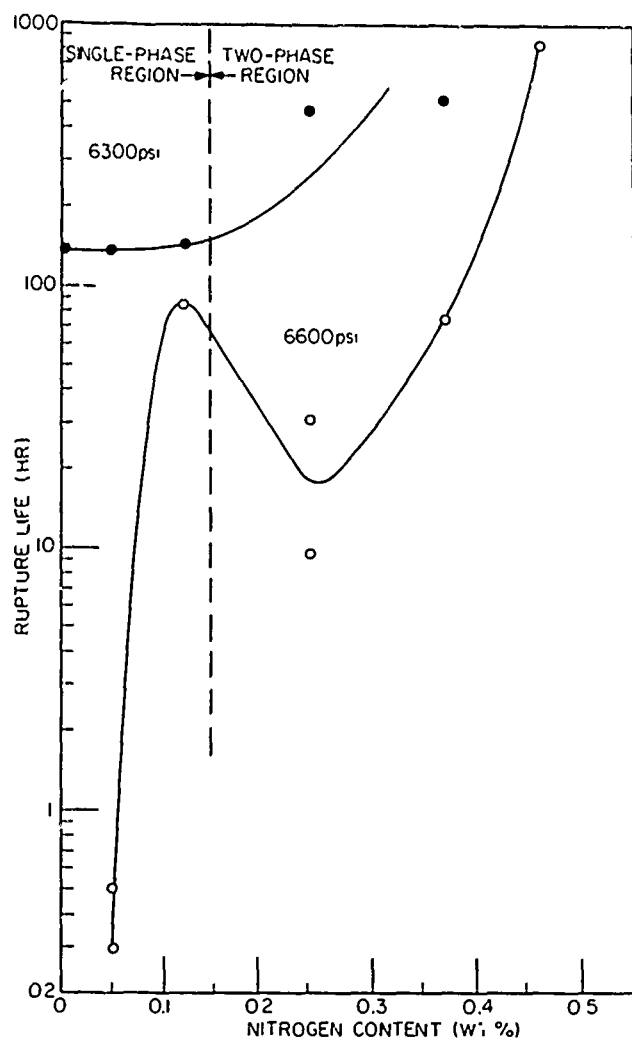


Fig. 9 - Influence of nitrogen on the rupture life of niobium at 1900° F

In the region of 1700° and 1900°F where probably intermediate-temperature creep mechanisms are controlling, rupture life was not greatly affected by a change in temperature. As shown in Table 2 for the Nb-0.12% N material, the stress to produce rupture in 88 hours was about the same, approximately 6500 psi, at the two temperatures cited. A similar behavior was noted for the Nb-0.24% N alloy at 6500 and 6300 psi. In contrast, a change in test temperature from 1900° to 2300°F, where high-temperature creep mechanisms prevail, would require an estimated decrease in stress to 1400 psi to give a life of approximately 88 hours. It should be mentioned that the pure niobium was sensitive to small changes of stress in the 6000 to 6600 psi range at 1700° and 1900°F and, as a consequence, yielded more than a normal amount of scatter in rupture life.

The influence of nitrogen on minimum creep rate (Table 2 and Fig. 10) was found to be consistent with its effect on rupture life. Generally, an improvement in rupture life by the addition of nitrogen was accompanied by a corresponding decrease in minimum creep rate.

The elongation-at-fracture values for the materials, listed in Table 2, were high, at least 30 percent at 1700° to 1900°F and 66 percent at 2300° to 2600°F. Nitrogen showed no apparent influence on the elongation values of either the single-phase or two-phase alloys. Considerable necking of the specimens occurred as indicated by reduction of area measurements which in all cases were greater than 80 percent. Microscopic examination of the ruptured specimens showed no evidence of voids or cracks.

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Table 2
Creep Rupture Data

Temp. (°F)	Stress (psi)	Nitrogen Content (wt-%)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation in 1.0 in. (%)	Structure*
1700	6500	0.005	2.5	6.94	46	SP
		0.05	>497.3	0.00043	--	SP
		0.05	>1914.7	0.00040	--	SP
		0.12	87.9	0.0052	30	TP
		0.24	576.1	0.0029	39	TP
		0.37	>2126.0	0.00045	--	TP
1900	6400	0.005	>572.5	0.0011	--	SP
	6600	0.005	70.4	0.013	53	SP
		0.05	0.3	--	52	SP
		0.05	0.5	13.6	33	SP
		0.12	85.5	0.0044	33	SP
		0.24	9.5	0.16	47	TP
		0.24	30.5	0.08	32	TP
		0.37	76.1	0.067	44	TP
		0.46	841.5	0.0043	35	TP
	6500	0.005	0.1	--	36	SP
	6400	0.005	132.9	--	--	SP
	6300	0.005	0.1	--	28	SP
		0.005	136.6	0.0054	34	SP
		0.005	197.8	0.0080	41	SP
		0.05	133.6	0.006	38	SP
		0.12	142.5	0.0063	45	SP
		0.24	461.5	0.0043	37	TP
		0.37	502.0	0.014	37	TP
	6000	0.005	260.3	0.0079	42	SP
2300	2000	0.005	17.0	1.78	75	SP
		0.005	18.0	1.66	96	SP
		0.05	9.7	2.98	76	SP
		0.05	7.0	3.99	69	SP
		0.12	9.9	2.83	67	SP
		0.24	10.9	2.69	67	SP
		0.37	62.2	0.40	74	TP
		0.46	206.0	0.13	109	TP
	1500	0.12	34.8	0.82	72	SP
	1300	0.005	100.8	0.26	77	SP
		0.005	99.8	0.32	84	SP
		0.05	128.8	0.21	75	SP
		0.12	135.5	0.14	92	SP
		0.24	93.9	0.33	93	SP
		0.37	406.0	0.044	73	TP
2600	1000	0.005	17.2	1.71	87	SP
		0.05	18.6	1.73	95	SP
		0.12	18.0	1.99	89	SP
		0.24	18.3	2.05	94	SP
		0.37	8.5	3.31	82	SP
		0.46	47.4	0.65	81	TP
	800	0.005	50.9	0.48	66	SP
		0.005	38.4	0.62	68	SP
		0.05	61.1	0.52	96	SP
		0.12	55.4	0.57	90	SP
		0.24	44.0	0.74	90	SP
		0.37	31.3	0.98	99	SP
		0.46	121.6	0.28	99	TP

*SP - Single-phase; TP - two-phase

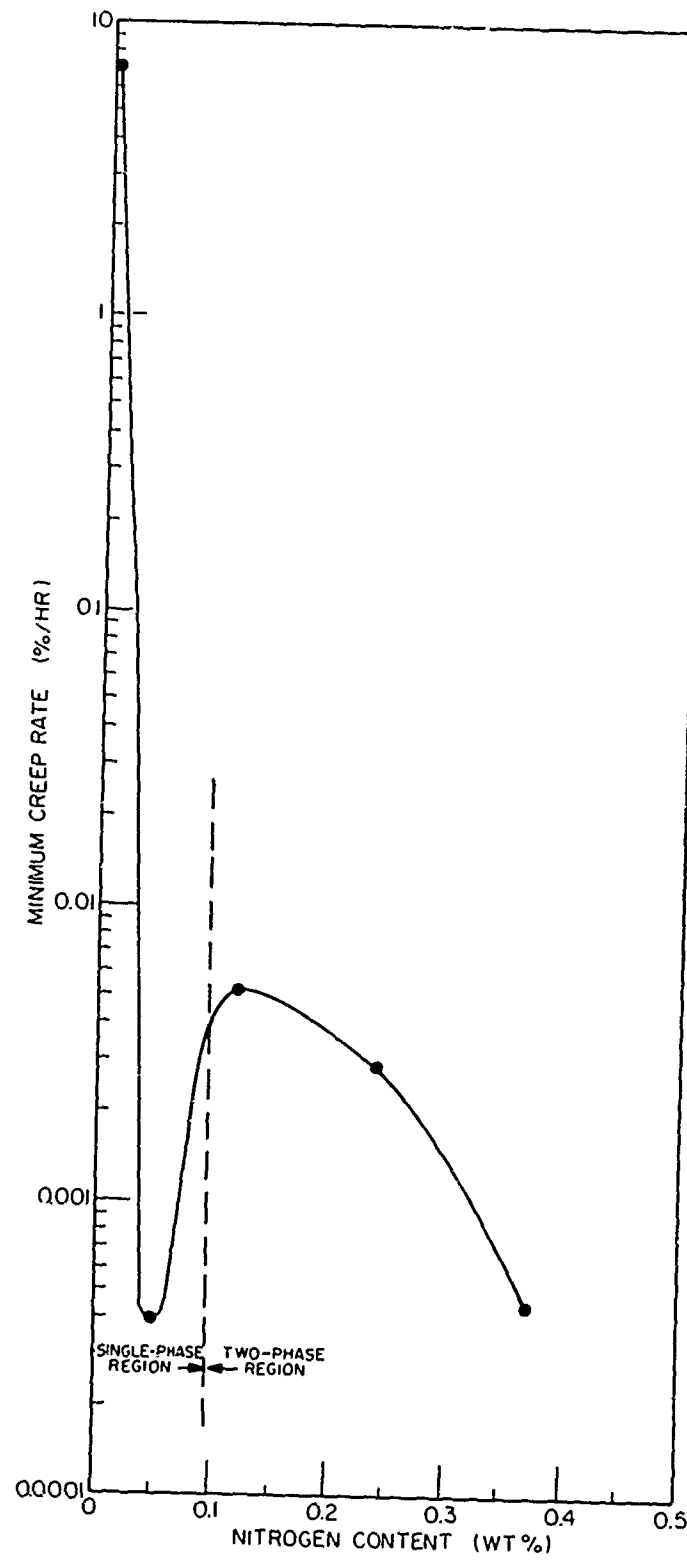


Fig. 10 - Influence of nitrogen on the minimum creep rate of niobium at 1700° F and 6500 psi

Creep-Curves — The microstructure of the niobium-nitrogen alloys apparently influenced the shape of the creep curves in the intermediate and high temperature regions. As illustrated in Fig. 11, single-phase alloys in the 1700° to 1900°F range show a very rapid acceleration on entering the third stage of creep, whereas the two-phase alloys are characterized by a gradual acceleration. The third stage of creep comprises a considerably larger proportion of the creep curves on the basis of time for the two-phase alloys than it does for the single-phase alloy.

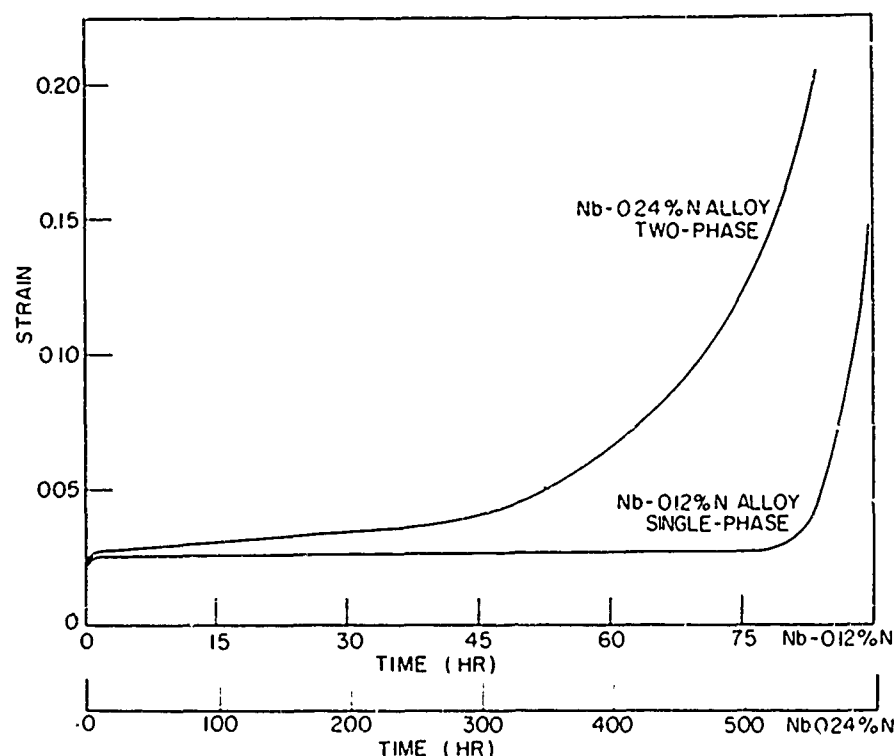


Fig. 11 - Creep curves of niobium-nitrogen alloys at 1700°F and 6500 psi showing the effect of microstructure on the shape of the curve.

At 2300° and 2600°F the single-phase alloys showed the usual three-stage creep behavior, while the two-phase alloys had essentially no primary creep (Fig. 12).

DISCUSSION

The strengthening of niobium by nitrogen in solid solution which takes place below 1900°F (approximately 0.5 melting temperature T_m), but not above, appears to be related to a corresponding change in the mechanism controlling creep. Usually in pure metals, flow is controlled by one mechanism at intermediate temperatures below 0.5 T_m and by another mechanism above 0.5 T_m . In body-centered cubic metals, the most likely rate-controlling mechanism at intermediate temperatures is the interaction of dislocations and interstitials, while at high temperatures it is dislocation climb (8).

The influence of nitrogen on creep-rupture strength observed in this study is what might be expected if these mechanisms are operating. Nitrogen, by interacting with dislocations, would strengthen niobium below 1900°F. Above this temperature, nitrogen diffuses rapidly in niobium and would have no effect on the dislocation climb process.

Thus at the higher temperatures creep and rupture are not affected by the presence of nitrogen.

Both of these mechanisms were apparently operating at 1900°F. At a stress of 6600 psi, nitrogen produced strengthening, but at 6300 psi, it had no effect. This behavior is consistent with the frequently made observation that lower stress corresponds to higher temperature in the way it affects creep.

In addition to the solid solution effects, the presence of a precipitate in the structure of the alloys generally produced strengthening at elevated temperatures, as would be expected. An exception was the creep behavior at 1700°F and for high stress at 1900°F, where the first introduction of a precipitate by increasing the nitrogen content caused a reduction in rupture life. A possible explanation for this behavior might be that the matrix of the two-phase alloy was softer than the single-phase alloy, as will be discussed later for a similar effect in room-temperature hardness.

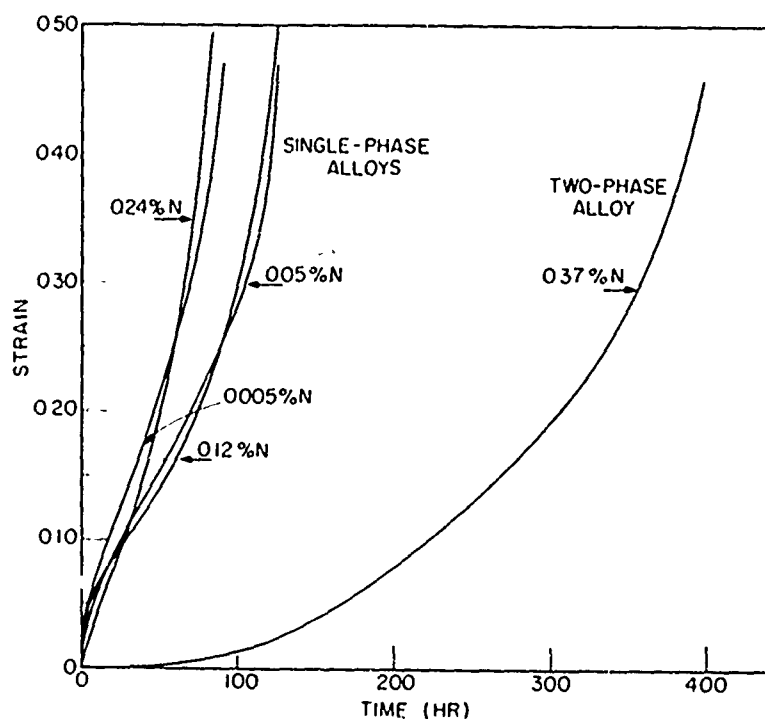


Fig. 12 - Creep curves of niobium-nitrogen alloys at 2300°F and 1300 psi showing the difference in creep behavior between single-phase and two-phase alloys

The influence of nitrogen on creep-rupture strength observed in this study is different in some respects from that of oxygen reported previously (1). At temperatures above $0.5 T_m$, oxygen in solution strengthens niobium while nitrogen does not. This disparity in behavior is inconsistent with the expectation that, if a difference in the effects of the two interstitials on strength should exist, the nitrogen, because of its lower mobility and higher activation energy for diffusion would retain its strengthening effect to higher temperatures than the oxygen. Inasmuch as the influence of nitrogen conforms to theoretical considerations, that of oxygen may be considered to be unusual. Perhaps oxygen forms complex clusters with substitutional impurities in the niobium, as has been suggested (9), and thus strengthens the material at high temperatures.

Considerable strengthening at room temperature was obtained by nitrogen in solution, but the introduction of the nitride phase in the structure did not enhance the strength significantly. In some instances the appearance of the nitride phase was actually accompanied by a decrease in hardness of the alloy, in comparison to the nearest single-phase alloy. This phenomenon was evident only when the alloys were cooled from the homogenization temperature at the slowest rate, thus indicating a critical dependence upon cooling rate. To account for this effect, it can be deduced that, since the nitride is very hard, the matrix of the two-phase structure is softer than the single-phase alloy. It is possible that the matrix of the two-phase structure, because of the precipitation effect, contains less nitrogen than does the single-phase alloy which would be supersaturated. Precipitates would serve to attract solute nitrogen atoms, thus tending to prevent supersaturation of the matrix. The hardness contribution of the nitride particles in the two-phase structure evidently does not sufficiently compensate for the reduced hardness of the matrix; consequently, an overall softer structure is realized.

This effect on room-temperature hardness might be reflected in the unusual creep behavior at 1700° and 1900°F mentioned previously. The difference in nitrogen content between the single-phase alloys and the matrix of the two-phase alloy evident at room temperature might be retained to these elevated temperatures and thus cause the two-phase alloy to be weaker than the single-phase alloy in the region of the phase boundary.

SUMMARY

To determine the effect of nitrogen on the mechanical behavior of niobium, the metal with additions of nitrogen up to 0.46 percent was tested in tension and creep rupture at various temperatures to a maximum of 2600°F. The principal results of the investigation were the following.

1. Nitrogen in solid solution increased the room-temperature hardness and tensile strength of niobium. At concentrations to give two-phase structures, nitrogen provided no additional increase in these properties.
2. Nitrogen raised the tensile strength of niobium at 1900°F, but at 2600°F raised it only at the higher concentrations.
3. Rupture life and creep resistance at 1700° to 1900°F were improved by nitrogen in solid solution, but at higher temperatures to 2600°F, they were not influenced by it. Higher nitrogen concentrations which produced two-phase microstructures improved these properties at all test temperatures.
4. Two-phase niobium-nitrogen alloys in creep at 1700° and 1900°F had a substantially greater proportion of their lives in third-stage creep than did the single-phase alloys. At 2300° and 2600°F, the single-phase alloys exhibited normal creep behavior, but the two-phase alloys had very little primary creep.
5. There was no influence of nitrogen on ductility at elevated temperatures; the elongation-at-fracture values for the tension and creep-rupture specimens were above 39 percent. Brittleness was evident only at room temperature in the 0.37 percent nitrogen alloy.
6. In the alloys that were slow cooled, the room-temperature hardnesses of the two-phase niobium-nitrogen alloys were lower than the highest nitrogen (0.05 percent) single-phase alloy, even though the nitride phase was very hard.

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13 ABSTRACT <p>Variations in the mechanical properties of niobium caused by nitrogen additions in amounts to give single-phase and two-phase structures were investigated at both room and high temperatures. Nitrogen in solid solution raised room-temperature hardness and tensile strength. Increasing the nitrogen content to give two-phase structures however produced no additional strengthening. At elevated temperatures, the effect was different in that, with increase in nitrogen content, there was a continued rise in tensile strength through the two-phase region.</p> <p>Creep-rupture strength of niobium was improved by nitrogen in solution at temperatures slightly below 0.5 times the melting temperature, whereas above this temperature the strength was unaffected. This change in the nitrogen effect with temperature appears to be related to a concurrent change in the mechanism controlling creep. An increase in the nitrogen content of the two-phase alloys produced strengthening throughout the 1700° to 2600°F test range. Ductility was high for all alloys at elevated temperatures and, except for a high-nitrogen alloy, also at room temperature.</p>			

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